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LUBRICATION OF TITANIUM SURFACES MODIFIED BY  
METALLIC DIFFUSION

by

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(6) LUBRICATION OF TITANIUM SURFACES MODIFIED BY  
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## Lubrication of Titanium Surfaces Modified by Metallic Diffusion

By ARTHUR SHAPIRO<sup>1</sup> and HENRY GISSER (ASLE)<sup>2</sup>

Lubrication of titanium by surface modification consisting of diffusion of other metals into the surface was studied. Coatings, approximately 0.001 in., of chromium, cobalt, copper, gold, iron, nickel, platinum, and silver were electroplated on titanium, followed by diffusion at 860 C and 10-20 mm Hg. Tin and lead were diffused by immersing the titanium in molten baths of these metals. Diffusion bands of approximately 10 to 15 thousandths of an inch were obtained.

Coefficient of friction measurements were made at various depths using a series of lubricants containing fatty acids, chloride, sulphide, and phosphite additives. The friction coefficient was reduced from about 0.25, for unlubricated titanium, to 0.06-0.10 in many instances, depending on the metal lubricant combination.

Loads of 20 to 40 Kg were sustained by some of the modified surfaces on the Four Ball Tester and 2500 to 3000 pounds on the Falex Machine. With pure titanium, gross seizure occurred at a load of 2 Kg on the Four Ball and 250 pounds on the Falex Machine.

### Introduction

TITANIUM as a pure metal is unusually strong, and because it has a specific gravity only a little over half that of steel, it has the advantage of an exceptionally high strength weight ratio. Another great advantage is its corrosion resistance, which places titanium in the same class as stainless steels.

The application of titanium as a structural material has, however, been beset with difficulties. Many problems have been encountered both in the manufacture and fabrication of titanium and its alloys. The machining of this material alone has raised many involved and interesting problems. In certain applications the surface properties of titanium need to be improved. These applications include joinability to other metals (soldering and adhesive bonding), thermal conductivity, electrical conductivity, oxidation resistance at elevated temperatures, and what is of particular concern here, wear and galling resistance, and the improvement of frictional properties.

Wear is a general term and Alman (1) has proposed that it consists of three general processes: the loss of metal from one or both rubbing surfaces, the transfer of metal from one surface to another, and the displacement of metal by plastic flow. Titanium is particularly susceptible to the second type of wear—the transfer of

metal from one surface to another. The smearing of titanium parallels that encountered with aluminum during the early days of experimenting with the latter. That difficulty was eventually cured by alloying, heat treatment, conversion coatings, electroplating of other metals, and the use of proper lubricants. The present difficulties with titanium may be expected to be overcome by any of the above or combination of the above methods.

A large amount of research has gone into a search for lubricants for titanium to reduce wear and the high coefficient of friction. This high coefficient of friction is caused by ready transfer of titanium to the mating surfaces, with the result that wear and friction characteristics are poor. Rabinowicz (2) has shown that many of the more commonly used lubricants are totally ineffective when used on titanium. Only a few lubricants produced a marked decrease in coefficient of friction from that of unlubricated titanium. The coefficient of friction of unlubricated surfaces was 0.49, of polyethylene glycols 0.26, and of polychlorotrifluoroethylene (pour point —54 C) 0.21. However, no good lubrication was brought about. The difficulty with titanium is that no strong natural bonding or reaction with its surface takes place.

All metals tend to gall to some extent, but usually some combination of lubricant and/or mating surface can be found which overcomes this wear. To date, there has been relatively scant success in finding combinations of metal surface and lubricant which will give satisfactory service against bare titanium. As in the instance of electroplating, the development of successful lubricants has not to date provided satisfactory lowering of the coefficient of friction and reducing wear.

Inorganic coatings chemically and electrochemically produced on titanium have provided good wear resistance

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(3) for some applications (e.g., metal forming). The chemical conversion coatings such as the phosphate and fluoride are applied by immersion of the work in the proper solution. Anodized surfaces are produced by making titanium the anode in a particular electrolyte. These coatings help eliminate the metal-to-metal contact that causes seizing, and also improve the surface retention of lubricants.

An approach toward solving the titanium lubrication problem is the alloying of the titanium surfaces to make it receptive to lubrication by conventional lubricants. A great deal of research has been done on the alloying of titanium with other metals. The position of titanium among the other metals of the Periodic System is very favorable for alloying. Practically all of the common and most useful metals have interatomic distances within 15% of that of titanium. This is important in that the metals within this 15% atomic diameter range have a size factor that is favorable for forming considerable solid solutions with titanium by atomic substitution (4). In addition, some of the elements of small atomic size, as carbon and hydrogen, form solid solutions interstitially. In general, then, this leaves only a few metals of comparatively large atom size, the rare earths and most of the alkaline and alkaline earth metals, as being unpromising alloying constituents from the standpoint of atomic size and solid solubility. Based upon the theoretically favorable alloying ability of titanium with many other metals, it appeared that if a relatively adherent electroplate could be deposited upon titanium, proper diffusion techniques would produce an alloy concentration gradient in the surface, and the resultant modified surface could then be studied to determine its friction and wear behavior. Some work in this direction has been reported (5) in an attempt to improve lubrication with titanium gears. Both copper and aluminum were tried as surface modifiers by plating and diffusion, but the attempts were unsuccessful in solving of the lubrication problem.

Considerable work has been done toward developing methods for electroplating adherent metal coatings on titanium. The development of successful methods, however, has not kept pace with the needs. Some of the procedures for activating and plating titanium are described in the literature (6-9). (Other methods, not published because of proprietary interests, are reported as successful.)

#### Surface modification

##### ELECTROPLATING

Metals were electroplated on 75A commercially pure titanium having the following properties: Tensile yield strength, 76,700 psi; ultimate tensile strength, 87,200 psi; elongation, 23.5%; C, .08%; N, .02%; H, .0050%. The specimen size was  $2\frac{1}{2} \times 1\frac{1}{2} \times \frac{1}{4}$  in. Specific procedural details for each metal electroplated are given

in the Appendix. Lead and tin coatings were obtained by diffusing the titanium in the molten metal.

##### DIFFUSION

Diffusion was conducted in a vacuum furnace at  $10^{-6}$  Torr. The specimens were placed in the furnace, which was then evacuated, and the temperature raised to 860 C for 7 hr, after which the heat was shut off, and the specimens cooled for approximately 18 hr, under vacuum, and were then removed from the furnace at approximately 60 C. Lead and tin were diffused during immersion in the molten metal at 860 C for 7 hours.

##### NATURE OF THE MODIFIED SURFACE

The depth to which diffusion had been accomplished was determined by Knoop hardness measurements of sectioned specimens (previously polished successively on 1G, 400-A, 600-A, 3/0 and 4/0 grit papers). Hardness measurements were carried out on a micro hardness tester using the Knoop indenter. Studies have indicated that with this indenter, the major part of the elastic recovery of the impression upon removal of the load takes place crosswise rather than along the length of the impression. Consequently, a very close measurement of the unrecovered indented area can be obtained by taking the measurement of the long diagonal only. A load of 500 gm and a magnification of  $\times 20$  was used in making all measurements. Three distinct patterns of hardness were found, depending upon the nature of the diffused metal. With lead, gold, platinum, and iron, the hardness closest to the surface was low and gradually increased until the hardness of titanium was reached. With copper, silver, cobalt, chromium, and nickel, the hardness increased and then gradually decreased to the hardness of titanium. With tin the hardness decreased with increasing depth until it reached the hardness of titanium. Unplated titanium gave the same pattern as tin after vacuum heating. The depths metals diffused into titanium are given in Table 1.

TABLE 1  
Depth of Metal Diffused  
into the Titanium Surface

Metal diffused	Depth (inch)
Chromium	0.009
Cobalt	0.012
Copper	0.012
Gold	0.007
Iron	0.010
Lead	0.015
Nickel	0.007
Platinum	0.005
Silver	0.010
Tin	0.009

##### Coefficient of friction

Friction experiments on the modified titanium surfaces were conducted at room temperature using a Bowden-

Leben type apparatus. Coefficients of friction were determined at two levels in the diffused layer. The surface was ground to depths given in Table 2. The rider used was a 52100 steel ball and the coefficients of friction were determined with cetane and with a solution of 1% palmitic acid in cetane. The applied load was 100 gm. The data are given in Table 2, each entry being the mean of five measurements.

TABLE 2  
Coefficient of Friction Measurements

Metal	Total depth of grind (in.)	Lubricated with cetane	Lubricated with 1% palmitic acid in cetane
Copper	0.004	0.19 <sup>a</sup>	0.12 <sup>a</sup>
	0.006	0.19 <sup>a</sup>	0.22 <sup>a</sup>
Nickel	0.003	0.09 <sup>b</sup>	0.07 <sup>b</sup>
	0.006	0.15 <sup>a</sup>	0.07 <sup>a</sup>
Tin	0.004	0.22 <sup>a</sup>	0.16 <sup>a</sup>
	0.006	0.18 <sup>a</sup>	0.17 <sup>a</sup>
Silver	0.001	0.09 <sup>b</sup>	0.06 <sup>b</sup>
	0.0015	0.07 <sup>b</sup>	0.06 <sup>b</sup>
Lead	0.004	0.17 <sup>a</sup>	0.15 <sup>a</sup>
	0.007	0.23 <sup>a</sup>	0.19 <sup>a</sup>
Cobalt	0.004	0.22 <sup>a</sup>	0.16 <sup>a</sup>
	0.0120	0.12 <sup>a</sup>	0.05 <sup>b</sup>
Platinum	0.001	0.18 <sup>a</sup>	0.17 <sup>a</sup>
	0.0015	0.15 <sup>a</sup>	0.05 <sup>b</sup>
Gold	0.0025	0.10 <sup>a</sup>	0.09 <sup>b</sup>
	0.003	0.20 <sup>a</sup>	0.17 <sup>a</sup>
Iron	0.002	0.17 <sup>a</sup>	0.16 <sup>a</sup>
	0.003	0.15 <sup>a</sup>	0.09 <sup>a</sup>
Chromium	0.002	0.06 <sup>b</sup>	0.05 <sup>b</sup>
	0.003	0.07 <sup>b</sup>	0.06 <sup>b</sup>
Unmodified	0.001	0.21 <sup>a</sup>	0.15 <sup>a</sup>
Titanium	0.002	0.16 <sup>a</sup>	0.12 <sup>a</sup>

<sup>a</sup> Stick-slip.

<sup>b</sup> Smooth sliding.

At the first depth, copper, gold, tin, platinum, lead, cobalt, and iron exhibited stick-slip. Chromium, silver, gold, and nickel gave smooth sliding with low coefficients of friction (below 0.10, palmitic acid in cetane lubricant). At the second depth, nickel, silver, cobalt, platinum, iron and chromium gave low coefficients of friction (below 0.10, palmitic acid in cetane), but of these, smooth sliding (no observable stick-slip) was obtained only with silver, cobalt, platinum, and chromium.

To study higher alloy concentrations and greater depths, friction measurements were made on titanium surfaces plated with approximately 0.006 in. thick nickel and iron followed by diffusion. The data are shown in Table 3. With iron, at low depths, sliding was smooth. Deeper, where the titanium concentration is higher, sliding was smooth only with palmitic acid. This indicated that the layer was sufficiently rich in iron or an iron-titanium phase to produce adequate chemical reaction with palmitic acid. Further progress into zones richer in titanium produced stick-slip indicative of poor frictional qualities. With nickel, the frictional results in the various zones approximated that of iron.

Further experiments were conducted to determine the effectiveness of conventional lubricant additives on the modified titanium surface. The compounds, studied at a concentration of 1% in cetane, were tricresyl phosphate, diisopropyl phosphite, hexachloroethane, 42% chlorinated paraffin, octyl mercaptan and benzyl disulphide. The average of five friction determinations are shown in Table 4.

Lead modified surfaces produced stick-slip with all six lubricants. Cobalt yield smooth sliding only with hexachloroethane. The chromium modified surface showed improved lubrication over that of titanium alone with diisopropyl phosphite, although the friction traces were somewhat irregular. Copper, which was plated about 0.002 in. thick, was diffused at what happened to be the copper-titanium eutectic point (860 C) and vigorous dif-

TABLE 3  
Coefficient of Friction on Iron and Nickel Modified Titanium Surfaces

Total depth of grind (in.)	Iron		Nickel		Titanium As Received		Titanium—7 hours at 860 C in vacuum	
	Lubricated with cetane	Lubricated with 1% palmitic acid in cetane	Lubricated with cetane	Lubricated with 1% palmitic acid in cetane	Lubricated with cetane	Lubricated with 1% palmitic acid in cetane	Lubricated with cetane	Lubricated with 1% palmitic acid in cetane
0	—	—	0.11 <sup>a</sup>	0.08 <sup>a</sup>	—	—	—	—
0.002	0.10 <sup>a</sup>	0.06 <sup>a</sup>	0.18 <sup>b</sup>	0.09 <sup>a</sup>	0.18 <sup>b</sup>	0.11 <sup>b</sup>	0.25 <sup>b</sup>	0.14 <sup>b</sup>
0.006	0.09 <sup>a</sup>	0.06 <sup>a</sup>	0.15 <sup>b</sup>	0.15 <sup>b</sup>	0.21 <sup>b</sup>	0.11 <sup>b</sup>	0.25 <sup>b</sup>	0.23 <sup>b</sup>
0.010	0.28 <sup>b</sup>	0.08 <sup>a</sup>	0.29 <sup>b</sup>	0.26 <sup>b</sup>	0.17 <sup>b</sup>	0.17 <sup>b</sup>	0.29 <sup>b</sup>	0.22 <sup>b</sup>
0.014	0.22 <sup>b</sup>	0.15 <sup>b</sup>	0.22 <sup>b</sup>	0.22 <sup>b</sup>	0.19 <sup>b</sup>	0.22 <sup>b</sup>	0.21 <sup>b</sup>	0.17 <sup>b</sup>
0.018	0.15 <sup>b</sup>	0.18 <sup>b</sup>	0.24 <sup>b</sup>	0.19 <sup>b</sup>	0.20 <sup>b</sup>	0.14 <sup>b</sup>	0.23 <sup>b</sup>	0.18 <sup>b</sup>

<sup>a</sup> Smooth sliding.

<sup>b</sup> Stick-slip.



TABLE 4  
Coefficient of Friction with Various Lubricants

Metals	Total depth of grind (in.)	Tricresyl phosphate	Octyl mercaptan	42% Chlorinated paraffin	Benzyl disulphide	Hexachloroethane	Diisopropyl phosphite
Chromium <sup>a</sup>	0.0005	0.27 <sup>b</sup>	0.25 <sup>b</sup>	0.16 <sup>b</sup>	0.21 <sup>b</sup>	0.22 <sup>b</sup>	0.10 <sup>c</sup>
	0.001	0.32 <sup>b</sup>	0.18 <sup>b</sup>	0.24 <sup>b</sup>	0.27 <sup>b</sup>	0.28 <sup>b</sup>	0.14 <sup>c</sup>
	0.0015	—	—	—	—	—	0.22 <sup>b</sup>
Cobalt	0.001	0.20 <sup>b</sup>	0.21 <sup>b</sup>	0.21 <sup>b</sup>	0.22 <sup>b</sup>	0.10 <sup>c</sup>	0.21 <sup>b</sup>
	0.002	0.22 <sup>b</sup>	0.20 <sup>b</sup>	0.22 <sup>b</sup>	0.22 <sup>b</sup>	0.25 <sup>b</sup>	0.20 <sup>b</sup>
Copper	0.001	0.10 <sup>c</sup>	0.06 <sup>c</sup>	0.20 <sup>b</sup>	0.09 <sup>c</sup>	0.08 <sup>c</sup>	0.11 <sup>c</sup>
	0.002	0.26 <sup>b</sup>	0.25 <sup>b</sup>	0.21 <sup>b</sup>	0.24 <sup>b</sup>	0.22 <sup>b</sup>	0.09 <sup>c</sup>
	0.003	—	—	—	—	—	0.28 <sup>b</sup>
Gold	0.0005	0.09 <sup>c</sup>	0.09 <sup>c</sup>	0.09 <sup>c</sup>	0.08 <sup>c</sup>	0.09 <sup>c</sup>	0.11 <sup>c</sup>
	0.0015	0.09 <sup>c</sup>	0.12 <sup>c</sup>	0.21 <sup>b</sup>	0.19 <sup>b</sup>	0.12 <sup>c</sup>	0.09 <sup>c</sup>
	0.0025	0.28 <sup>b</sup>	0.29 <sup>b</sup>	0.26 <sup>b</sup>	0.21 <sup>b</sup>	0.24 <sup>b</sup>	0.24 <sup>b</sup>
Iron	0.001	0.09 <sup>c</sup>	0.10 <sup>c</sup>	0.09 <sup>c</sup>	0.23 <sup>b</sup>	0.21 <sup>b</sup>	0.21 <sup>b</sup>
	0.002	0.18 <sup>b</sup>	0.18 <sup>b</sup>	0.26 <sup>b</sup>	0.20 <sup>b</sup>	0.22 <sup>b</sup>	0.20 <sup>b</sup>
Nickel	0.001	0.10 <sup>c</sup>	0.28 <sup>b</sup>	0.10 <sup>c</sup>	0.09 <sup>c</sup>	0.10 <sup>c</sup>	0.09 <sup>c</sup>
	0.002	0.22 <sup>b</sup>	0.27 <sup>b</sup>	0.14 <sup>b</sup>	0.30 <sup>b</sup>	0.28 <sup>b</sup>	0.21 <sup>b</sup>
Platinum	0.0005	0.11 <sup>c</sup>	0.17 <sup>b</sup>	0.21 <sup>b</sup>	0.14 <sup>c</sup>	0.09 <sup>c</sup>	0.11 <sup>c</sup>
	0.001	0.19 <sup>b</sup>	0.12 <sup>b</sup>	0.14 <sup>b</sup>	0.17 <sup>b</sup>	0.19 <sup>b</sup>	0.12 <sup>c</sup>
	0.0015	0.24 <sup>b</sup>	0.25 <sup>b</sup>	0.23 <sup>b</sup>	0.28 <sup>b</sup>	0.24 <sup>b</sup>	0.26 <sup>b</sup>
Silver	0.005	0.11 <sup>c</sup>	0.20 <sup>b</sup>	0.13 <sup>c</sup>	0.17 <sup>b</sup>	0.21 <sup>b</sup>	0.08 <sup>c</sup>
	0.0015	0.25 <sup>b</sup>	0.21 <sup>b</sup>	0.21 <sup>b</sup>	0.21 <sup>b</sup>	0.19 <sup>b</sup>	0.22 <sup>b</sup>
Lead	0.001	0.20 <sup>b</sup>	0.20 <sup>b</sup>	0.21 <sup>b</sup>	0.21 <sup>b</sup>	0.22 <sup>b</sup>	0.18 <sup>b</sup>
	0.002	0.25 <sup>b</sup>	0.21 <sup>b</sup>	0.22 <sup>b</sup>	0.25 <sup>b</sup>	0.17 <sup>b</sup>	0.22 <sup>b</sup>
Unmodified	0.001	0.22 <sup>b</sup>	0.26 <sup>b</sup>	0.24 <sup>b</sup>	0.26 <sup>b</sup>	0.25 <sup>b</sup>	0.23 <sup>b</sup>
Titanium	0.002	0.22 <sup>b</sup>	0.22 <sup>b</sup>	0.21 <sup>b</sup>	0.27 <sup>b</sup>	0.24 <sup>b</sup>	0.24 <sup>b</sup>

<sup>a</sup> Plating thickness of 0.0004 in.

<sup>b</sup> Stick-slip.

<sup>c</sup> Smooth sliding.

fusion had occurred in both directions. Grinding to a depth of 0.001 in. showed a diffused silvery copper surface which produced good frictional conditions with tricresyl phosphate, octyl mercaptan, benzyl disulphide, hexachloroethane, and diisopropyl phosphite. Removal of another layer of 0.001 in. produced a surface giving good frictional properties with diisopropyl phosphite alone.

Gold, which was plated to a thickness of 0.001 in., was like copper in that on grinding to a depth of 0.0005 in., a silvery gold surface was obtained. This layer produced low coefficients of friction with all six lubricants. Removal of another 0.001 in. produced surfaces which were well lubricated by tricresyl phosphate, octyl mercaptan, hexachloroethane, and diisopropyl phosphite. Iron ground midway into the plated layer produced a surface giving good frictional results with tricresyl phosphate, octyl mercaptan, and 42% chlorinated paraffin.

Nickel ground to a depth of 0.001 in. gave a surface producing good frictional properties with all of the lubricants except octyl mercaptan. Platinum, ground to 0.0005 in. resulted in a surface which gave good frictional results with tricresyl phosphate, hexachloroethane, and diisopropyl phosphite and irregular friction traces with benzyl disulphide. Removal of another 0.0005 in. produced a surface which gave irregular friction traces

with diisopropyl phosphite. Silver, ground to a depth of 0.0005 in., produced smooth friction traces with tricresyl phosphate and diisopropyl phosphite.

One of the interesting results is the good frictional properties obtained in the diffusion zones of such unreactive metals as platinum, and gold. Also of interest is the fact that diisopropyl phosphite yielded good lubrication with many of the alloy surfaces. Titanium, in the as received condition, gave consistently poor frictional properties with all the lubricants studied. Stick-slip conditions were always present. Generally, the good lubrication effects produced are quite specific, depending on the depth of the diffusion layer measured, and the lubricant used.

Experiments were also conducted using a titanium ball rider in addition to the titanium plate. Work was done with silver and gold modified surfaces which had been first plated with approximately 0.001 in. of metal, then diffused. Measurements were made with and without removing metal from the ball rider and plate surfaces. The results in Table 5 show that good frictional characteristics were obtained after the removal of 0.001 in. On further removal of metal, poor frictional results, as indicated by stick-slip, were obtained. It is interesting to note the coefficients of friction were high and the

TABLE 5  
Coefficient of Friction Measurements on Titanium  
Rider and Plate (Plating Thickness = 0.001 in.)

Metal	Total depth of grind (in.)		Cetane	Benzyl disulphide 1% in cetane	Diisopropyl phosphite 1% in cetane
	Rider	Plate			
Silver	None	None	0.15 <sup>a</sup>	0.15 <sup>a</sup>	0.12 <sup>a</sup>
	0.001	0.001	0.09 <sup>b</sup>	0.09 <sup>b</sup>	0.10 <sup>b</sup>
	0.001	0.002	0.15 <sup>a</sup>	0.25 <sup>a</sup>	0.14 <sup>a</sup>
Gold	None	None	0.25 <sup>a</sup>	0.24 <sup>a</sup>	0.24 <sup>a</sup>
	0.001	0.001	0.09 <sup>b</sup>	0.08 <sup>b</sup>	0.12 <sup>b</sup>
	0.001	0.002	0.26 <sup>a</sup>	0.27 <sup>a</sup>	0.21 <sup>a</sup>

<sup>a</sup> Stick-slip.

<sup>b</sup> Smooth sliding.

motion was not smooth when rubbing took place between surfaces from which no metal had been removed. Here there are two like soft metals. After grinding the surface layer off, the improvement is obtained. This is probably associated with increasing hardness and minor differences in alloy concentration.

#### Four ball tests

The four ball studies were conducted at room temperature (20–27°C) at 600 rpm for 1 hr at various loads. Both the upper and lower balls were ½ in. diam. 75A titanium which had previously been given an electroplate with the alloying metals to a depth of 0.001 in. followed by diffusion.

The unmodified titanium specimens all seized immediately at a 10 kg load. The gold modified specimens exhibited better load carrying capacity, those lubricated with cetane sustaining a load of 10 kg, and those lubricated with diisopropyl phosphite and benzyl disulphide sustaining a load of 20 kg. The silver modified specimens

TABLE 6  
Four Ball Wear Test on Modified Titanium Surfaces<sup>a</sup>

Metal	Load (kg)	Cetane	Diisopropyl phosphite 2% in cetane	Benzyl disulphide 2% in cetane
Unmodified titanium	10	immediate failure	immediate failure	immediate failure
Gold	10	0.52	0.96	0.55
	20	failed-20 min	0.62	0.58
	30	—	failed-15 min	failed-45 min
Silver	10	0.51	0.75	0.71
	20	0.59	1.08	0.52
	30	0.79	0.90	0.71
	40	failed-30 min	1.10	failed-40 min
	50	—	failed-20 min	—

<sup>a</sup> Figures are scar diameters in millimeters.

gave the best results. Those lubricated with cetane and benzyl disulphide sustained a load of 30 kg and those with diisopropyl phosphite sustained a load of 40 kg. Tests were run in duplicate and the results (averages) are shown in Table 6. Excellent reproducibility in failure times ( $\pm$  five minutes) was obtained. Scar diameter reproducibility was not as good.

#### Falex machine tests

The Falex tests were conducted using jaw blocks made from FS 2320 steel and pins made from 75A titanium. The jaw load was increased from zero to 250 pounds by engaging the eccentric arm of the ratchet wheel. When the jaw load reached 250 pounds the eccentric arm was disengaged and the load maintained for 1 minute. Using the same procedure, the jaw load was increased in increments of 250 pounds until failure occurred.

In Table 7 are listed the wear load and the failure load. The wear load is that load at which the eccentric arm

TABLE 7  
Falex Wear Test on Modified Titanium Surfaces

Pin	Cetane		Diisopropyl phosphite 2% in cetane		Benzyl disulphide 2% in cetane	
	Wear (lb)	Failure (lb)	Wear	Failure	Wear	Failure
Unmodified titanium	0	0	0	0	0	0
Gold	2250	3000	1500	2750	2000	3000
Copper	2250	3000	1250	3000	1750	1750

needs to engage between 10 and 15 teeth on the ratchet wheel in order to regain the original applied load. Since each tooth corresponds to 0.000556 in. of wear, this is equivalent to 0.000556 in. to 0.000834 in. of wear. The failure load is that load at which either seizure occurs between the pin and the blocks or the brass pin holding the pin specimen between the blocks ruptures. The unmodified titanium pins failed immediately, whereas the diffused pins sustained loads of 1750 to 3000 pounds. Rate of wear and cumulative amount of wear in the Falex Machine tests are plotted in Figs. 1–6. In every instance there was a fairly high initial wear rate which immediately decreased and then increased again with increasing load. The initial high wear rate was due to the soft outer surface in which the titanium concentration was very low. The wear rate curve is the resultant of increasing wear rate due to increasing load, and changing wear rate due to increase in the titanium concentration as the surface is worn to a progressively greater depth.

#### Discussion

The effectiveness in friction reduction varied with the metal-additive combination. In general, gold, nickel, copper, and platinum gave good results, iron and silver fair results, while cobalt and chromium were effective

with very few additives. Lead was not effective in friction reduction. Insufficient data are available on tin to indicate its effectiveness. While diisopropyl phosphite, tricresyl phosphate, and palmitic acid appeared to be the most effective of the additives, it is difficult to determine the degree of significance of the observed differences among additives. In almost every instance palmitic acid lowered the friction coefficient over that obtained with

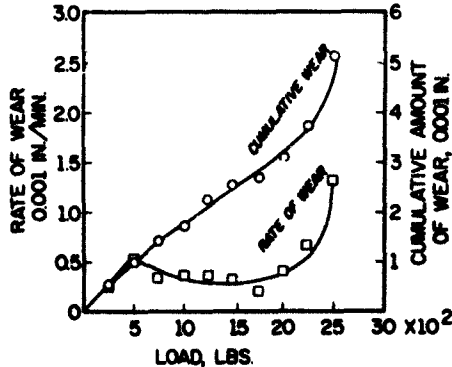


FIG. 1. Falex test on gold modified titanium surface with cetane lubricant.

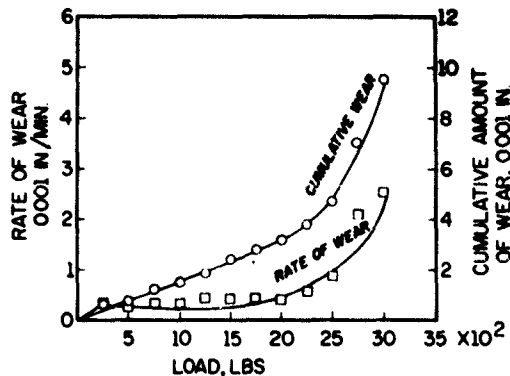


FIG. 2. Falex test on gold modified titanium surface with benzyl disulfide lubricant 2% in cetane.

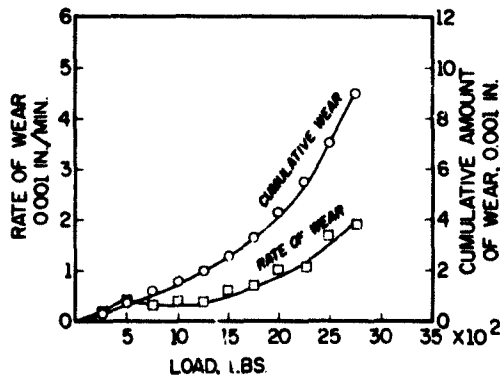


FIG. 3. Falex test on gold modified titanium surface with diisopropyl phosphite lubricant 2% in cetane.

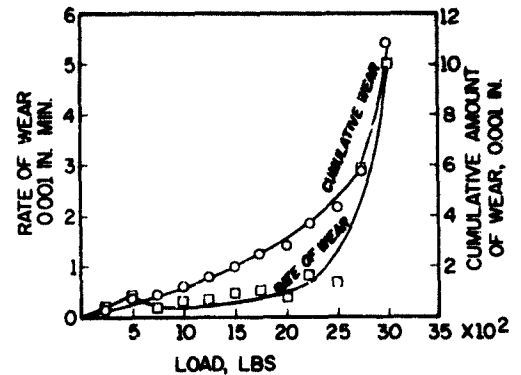


FIG. 4. Falex test on copper modified titanium surface with cetane lubricant.

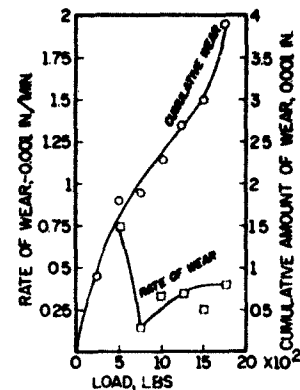


FIG. 5. Falex test on copper modified titanium surface with benzyl disulfide lubricant 2% in cetane.

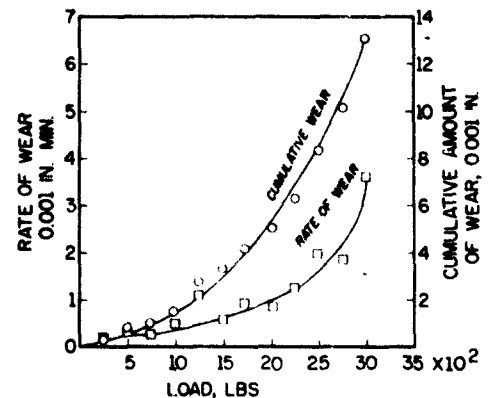


FIG. 6. Falex test on copper modified titanium surface with diisopropyl phosphite lubricant 2% in cetane.

cetane alone, but smooth sliding was not always obtained. (It should be noted that friction measurements with palmitic acid were run in most instances at considerable depths below the original surface, and results with palmitic acid are not always comparable with those obtained with other additives.)

The solubility of the modifying metals in the alpha phase of titanium at room temperature is quite low. At the eutectoid temperature (approx. 600–800 C for the metals studied) gold is soluble to the extent of approximately 10%, copper 2.1%, silver, cobalt, chromium, and iron 1% or less, and nickel nil. At room temperature, the solubilities would be considerably less. Observable diffused metal was obtained at depths from 0.005 to 0.015 in (Table 1). While the exact concentration gradients of the modifying metals are not known, it is reasonable to assume that at the depths where transitions in friction occurred with most metal-additive combinations, the concentration of modifying metal exceeded its solubility in the alpha phase of titanium. The effectiveness of the modifying metal is therefore probably not associated with its being in solution. Further work on the metallurgical structure of the modified surfaces is needed in order to clarify the mechanism of friction reduction.

In the Four Ball and Falex tests, strikingly different results were obtained in load carrying capacity between modified and unmodified titanium surfaces, independent of additive. Limited data on the modified surfaces indicate differences in results depending on additive. In the Four Ball test, the additives increased the load bearing capacity slightly, while in the Falex test the effect of the additives was in the other direction.

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#### Appendix

##### ELECTROPLATING PROCEDURES

**Chromium.** The specimen was degreased in toluene, alkaline cleaned, water washed, and pickled in a solution containing 185 ml of 48% hydrofluoric acid and

9 ml of concentrated nitric acid per liter of aqueous solution. Chromium was plated according to the method of Stanley and Brenner (6). The specimen was dried and suspended for 10 to 15 minutes in a solution of hydrofluoric acid in acetic acid at a concentration of 125 ml of 48% hydrofluoric acid per liter, without current. A slight amount of gassing occurred, which soon ceased, and a film was formed on the surface. A 60 cycle alternating current of 14 to 28 amps/ft<sup>2</sup> was then passed through the specimens for 10 minutes. About 40 volts were required. The specimen was rapidly rinsed and transferred to a conventional chromic acid plating bath containing 400 gm of chromic acid and 4 gm of sulfuric acid per liter. The plating was done at 85 C and a current density of 1100 amps/ft<sup>2</sup>. Plating for 1 hour produced a plate 0.001 inches thick. A lead anode was used.

**Cobalt.** The specimen was degreased with toluene and sandblasted, using 60 mesh aluminum oxide, under a line pressure of 90 psi. The specimen was electroplated in a bath (7) containing 1893 gm of cobalt sulfate heptahydrate, 65 gm of sodium chloride and 170 gm of boric acid in 4 liters of solution, using a cobalt anode. The solution pH was 5. The plating was done at 35 C and a current density of 20 amp/ft<sup>2</sup>. Plating was continued for 2 hours to a thickness of 0.002 in.

**Copper.** The specimen was degreased with toluene, and pickled in a solution of 185 ml of 48% hydrofluoric acid, 25 ml conc. nitric acid and the remainder water to 1 liter. It was then rinsed in water and anodically etched (6) for 5 minutes at 24 amp/ft<sup>2</sup>. Instead of using acetic acid, a solution of 150 ml of 48% hydrofluoric acid in formic acid was used. Less heat was evolved using formic acid instead of acetic acid. The specimen was water rinsed, and plated in a copper fluoborate bath of the following composition: 448g/l of copper fluoborate, specific gravity 1.36, fluoboric acid to pH 0.6. Plating carried out at 50 amp/ft<sup>2</sup> produced a plate 0.002 inches thick at the end of 1 hour. A copper anode was used.

**Gold.** The specimen was toluene cleaned, and pickled in a solution of 185 ml of 48% hydrofluoric acid, 9 ml conc. nitric acid in 1 liter. After water rinsing, plating was carried out in a bath (8) containing 11 gm of potassium cyanide and 12.3 gm of potassium aurocyanide in a liter (distilled water), with a gold anode. Plating was carried out at a temperature of 60–70 C and a current density of 2 to 3 amp/ft<sup>2</sup>. Plating was continued for 2 hours to a thickness of 0.001 in.

**Iron.** The specimen was cleaned with toluene, and pickled in a solution of 900 ml 1:1 nitric acid, 100 gm ammonium bifluoride and 100 ml of fluorsilicic acid. After rinsing, plating was carried out in a bath (7) containing 375 gm of ferrous chloride and 138 gm of calcium chloride diluted to 1 liter with water, using an iron anode. The solution pH was 1. Plating temperature was 90 C and the current density was 20 amps/ft<sup>2</sup>. A plate thickness of 0.002 in. was obtained in 1 hour.

**Nickel.** The specimen was degreased in toluene, and

pickled in 185 ml of 48% hydrofluoric acid, 9 ml conc. nitric acid and the remainder water to 1 liter. After rinsing, the specimen was activated by placing for 5 minutes in a solution of 0.1 g/l of palladium chloride in water and then immersed in an acid type electroless nickel bath at a temperature of 82–86 C for ½ to 1 hour. Plating was carried out in a Watts type bath.

**Platinum.** After toluene cleaning, the specimen was placed in conc. hydrochloric acid (9) at a temperature of 90–100 C for 10 minutes, water rinsed, and plated in a bath containing 358 gm of ammonium nitrate (reagent grade), 45 gm of sodium nitrate (reagent grade), 13.8 gm of diammino platinous nitrite and 210 ml of 28% ammonium hydroxide (reagent grade) made up to 4 liters with distilled water, using a platinum anode. Plating was carried out at a temperature of 88–97 C and a current density of 10 amps/ft<sup>2</sup>. A plate thickness of 0.0004 in. was produced in 2 hours.

**Silver.** The sandblasted specimen was plated (7), first in a strike bath (14.7 gm silver cyanide and 238 gm sodium cyanide, technical grade in both cases, in 4 liters of solution) for 10 seconds at 21–30 C and current density of 20 amps/ft<sup>2</sup>; followed by plating in a bath containing 162 gm of silver cyanide, 224 gm of potassium cyanide and 179 gm of potassium carbonate in 4 liters of solution. Plating temperature was 20–30 C and current density was 4 amps/ft<sup>2</sup>. Silver anodes were used in both baths and a plate thickness of 0.0005 in. was obtained in 1 hour.

**Lead.** The specimen after degreasing in toluene and pickling in a solution of 900 ml of 1:1 nitric acid, 100 gm ammonium bifluoride, and 100 ml of fluosilicic acid, was rinsed, dried, and immersed in a molten lead bath at 860 C.

**Tin.** The specimen was pretreated as the lead specimen was, and immersed in a molten tin bath at 860 C.

## DISCUSSION

E. M. KOHN (Sun Oil Company, Marcus Hook, Pennsylvania):

The literature pertinent to the lubrication of Titanium contains a number of examples illustrating substantial friction reduction, as well as the elimination of galling, and seizure, between Titanium surfaces in rubbing contact. It is required that these surfaces be coated first with thin films of metals or salts. [See, for example (A1), (A2), and (A3).]

In no case, however, has the influence of thin metal coatings been so well detailed, and the sensitive dependence of friction and wear reduction on coating thickness been so effectively demonstrated, as in the present paper. The authors are to be congratulated for providing us with more than just a fine job. The effects shown are all the more remarkable in light of diffusion equations which predict a rapid reduction in concentration of diffused metal just below the Ti surface. Despite this, metals such as Pt and Au or Pb and Sn induce effects which contrast significantly to predictions made from experience at depths which are a substantial fraction of the overall diffusion depth.

The attention paid by the literature to mechanisms explaining these lubrication benefits has been scant. The present authors have made a start at this by recognizing the need to attack this problem from a metallurgical standpoint. Better knowledge of the compositions of metal phases existing near the surface after diffusion, and their properties, are essential in explaining these observations. Furthermore, such studies aimed at explaining the friction reducing properties of thin metal coatings may well contribute to newer concepts of the role played by boundary lubricants or their chemical products in more conventional situations. This aspect is dealt with more fully below.

Of the systems studied, coatings formed from Pb, Au, Pt, and Fe are examples of the general case treated by Bowden and Tabor (A4), where hard metals register improved lubrication properties when coated with a softer metal. An additional contributing factor, but more important in cases where surfaces harder than Ti coat the Ti, such as Sn, Cu, Ag, Co, Cr, and Ni, are the low-shear-strength films produced chemically between the lubricant and the surface coating.

Another role played by the metal coating and lubricant is to

reduce the formation of oxide and nitride films which high interface temperatures otherwise would produce, and which Miller and Holladay (A2) consider to be factors limiting the adhesion of lubricants to Ti, while not preventing galling and seizure.

Further, since cobalt-plated titanium out-performs heated titanium in friction reduction (see Table 2 and 3 respectively), after both are ground down to at least the diffusion depth of cobalt into titanium (see Table 1), where the cobalt is probably present only in solution, the writer is prompted to apply the results of some unpublished work (A5) and suggests a fourth factor contributing to the effectiveness of the various metal coatings to reduce friction.

In this work (A5) the loads to shear-fail metal were found to be strongly influenced by the surrounding organic liquid medium. In the present case then, alloys which melt at lower temperatures than titanium are believed to function by embrittling asperity welds made between titanium and other surfaces with which it is engaged under test conditions, i.e., lower melting alloys may promote the shear failure of asperities at reduced stress levels. Also, intermetallic compounds, which McQuillan and McQuillan (A6) show Ti to form with all of the deposited metals, may also embrittle asperity welds.

This effect, where a metal under applied stress can be severely embrittled by liquid metals, has been demonstrated in great detail by Rostoker, McCaughey and Markus (A7).

The writer would like to know how the experimental results are influenced by preparatory procedure, i.e., surface grinding technique, diffusion time, and diffusion temperature. Also, have the authors studied mixed metallic deposits in the form of initial thin layers of two or more metals plated on the surface of the Ti, and what is the effect of the coating on the tensile strength of titanium?

The writer suggests that other titanium-coated specimens besides cobalt be ground down to their respective diffusion depths, as defined by hardness measurements, and evaluated; and also that a more sensitive technique than hardness be used to judge diffusion depth. One method might be repeated measurement of the potential of the treated titanium specimen against a reference electrode during continued surface grinding until the potential equals that of untreated, but polished, titanium.

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## AUTHORS' CLOSURE:

The authors wish to express their appreciation to Dr. Kohn for his interesting and helpful commentary on their paper.

Dr. Kohn has pointed out a number of variables in the surface preparation techniques which may affect the results. Time did not permit a study of the effect of variation in surface grinding techniques, or diffusion time, or temperature. The surfaces were all

ground using a well-dressed 20-A-46-86VS wheel flooded with coolant to minimize surface distortion. Approximately 0.0003 inch of metal was removed with each pass of the wheel. With respect to diffusion, since the coefficient of mass diffusivity varies exponentially with absolute temperature, concentration gradients achieved in relatively short time at high temperature would require much longer times at lower temperatures. While all of the surfaces on which friction and wear measurements were made were diffused at 860 C for 7 hours, on some surfaces diffusion was allowed for 3 hours and well-defined diffusion gradients were obtained as indicated by hardness measurements and photomicrographs. It is to be expected that at a constant diffusion temperature, allowing sufficient time for some alloy gradient to be established, the lubrication properties would be improved, but the maximum depth at which good lubrication properties would be obtained would vary with the time of diffusion. A change in temperature makes for a more complex situation since any compounds formed during the diffusion process may be temperature dependent.

The study of diffusion of multiple metal systems is certainly desirable and further extension of this work should include exploration of such systems and study of the effect of the surface modification on other physical properties such as tensile strength.

Dr. Kohn's concept of the embrittlement of asperity welds as a step in the lubrication mechanism is very interesting and provides a reasonable explanation of the observed behavior of lubricants. Further work on this concept would be well-merited.

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- 4 - British Joint Services Mission  
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Washington, D. C.
- 1 - Southwest Research Institute  
Attn: Mr. R. Quillian  
Ordnance Fuels and  
Lubricant Laboratory  
8500 Culebra Road  
San Antonio 6, Texas
- 1 - Attn: Dr. P. M. Ku
- 1 - Commanding Officer  
Bureau of Ships  
Dept of the Navy  
Attn: Code 634A  
Washington 25, D. C.

<p>AD- FRANKFORD ARSENAL, Research and Development Group, Philadelphia 37, Pa. LUBRICATION OF TITANIUM SURFACES MODIFIED BY METALLIC DIFFUSION by Arthur Shapiro and Henry Gieser FA Report A63-2, Jan 1963; 15 pp incl tables &amp; illus OMS Code 5026, 11, 80100; DA Proj 593-21-060</p> <p>UNCLASSIFIED REPORT</p> <p>Lubrication of titanium by surface modification consisting of diffusion of other metals into the surface was studied. Coatings, approximately 0.001 in. of chromium, cobalt, copper, gold, iron, nickel, platinum, and silver were electroplated on titanium, followed by diffusion at 860 C and 10<sup>-5</sup>mm Hg. Tin and lead were diffused by immersing the titanium in molten baths of these metals. Diffusion bands of approximately 10 to 15 thousandths of an inch were obtained.</p> <p>Coefficient of friction measurements were made at various depths using a series of lubricants containing fatty acids, chloride, sulphide, and phosphite additives. The friction coefficient was reduced from about 0.25, for unlubricated titanium, to 0.06-0.10 in many instances, depending on the metal lubricant combination.</p> <p>DISTRIBUTION LIMITATIONS: None; obtain copies from ASTIA.</p>	<p>UNCLASSIFIED</p> <p>I. Lubricants 2. Lubrication 3. Titanium 4. Friction 5. Wear</p> <p>II. A63-2 Shapiro, Arthur Gieser, Henry OMS Code 5026, 11, 80100</p> <p>UNCLASSIFIED</p>
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AD-  
FA Report A43-2 (Cont'd)  
ACCESSION NO.

Leds of 20 to 40 Kg were examined by some of the  
modified surfaces on the Four Ball Tester and 2500 to  
3000 pounds on the Palm Machine. With pure titanium,  
gross volume occurred at a load of 2 Kg on the Four Ball  
and 250 pounds on the Palm Machine.

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